PROJECT DATA

Membrane Technology & Research Inc. - 02GO12057

Separation of Zein from Bioethanol Process Streams

Recipient:	Membrane Technology & Research, Inc.	Instrument Number:	DE-FG36-02GO12057
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Subcontractor(s):		B&R Number(s):	ED190602
		PES Number(s):	02-2140, 03-11015
EERE Program:	Biomass	State Congressional District	CA - 14

PROJECT SCOPE: The objective of this project is to develop an energy-efficient, low-cost membrane process to more effectively separate zein from the ethanol corn extract solution. A 12-month pilot-process design will be implemented to obtain enough data to optimize membranes to be selected for the proof-of-concept plant. A preliminary design of the plant will also be prepared. Concentration and purification of zein by ultrafiltration creates no new waste streams, but reduces energy consumption of spray-drying the zein/ethanol solution by 100,000 Btu/lb of zein produced.

FINANCIAL ASSISTANCE

Approved DOE Budget	$200,000	Approved DOE Share	$200,000
Obligated DOE Funds	$200,000	Cost Share	$24,153
Remaining Obligation	$0		
Unpaid Balance	$0	**TOTAL PROJECT**	**$224,153**

Project Period: 8/9/02-8/30/03

TECHNICAL PERFORMANCE
DE-FG36-02GO12057
Membrane Technology & Research Inc.
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PROJECT SYNOPSIS

Production of bioethanol from corn is only economically feasible because of a federal subsidy of $0.54/gal. The project will defray a significant portion of the cost of the process by producing zein, a 22,000 molecular weight corn protein, as a coproduct. Approximately 1 lb of zein can be produced for each gallon of bioethanol. Currently, zein costs $10-20/lb, so the total market is small. If the cost of producing zein can be reduced to about $1/lb, a large market will develop for the product as a biodegradable plastic, a food additive, and a paper-coating material. The key to low-cost zein production is an energy-efficient, low-cost membrane process to separate zein from the ethanol corn extract solution. A 12-month pilot-process design will be implemented to obtain enough data to optimize membranes to be selected for the proof-of-concept plant. A preliminary design of the plant will also be prepared. Concentration and purification of zein by ultrafiltration creates no new waste streams, but reduces energy consumption of spray-drying the zein/ethanol solution by 100,000 Btu/lb of zein produced.

Using, as an installed unit, basis a 100,000 gal/day dry milling bioethanol plant producing 100,000 lb/day of zein, the energy consumption for the proposed ultrafiltration unit is 0.85×10^9 Btu/day. In contrast, the energy consumption of a spray dryer performing the same function is 8.5×10^9 Btu/day.

SUMMARY OF TECHNICAL PROGRESS

In this project, MTR developed and demonstrated a process that produced a final zein product of 92% purity with a membrane that rejected over 99% of zein. A six-fold increase in zein feed concentration was achieved, producing concentrated zein for spray-drying. Analysis of the spraydried zein showed that most of the unfavorable odor and taste have been removed. MTR also identified an economical cleaning process to address the issue of membrane fouling.

During the course of the project, several spiral-wound membrane modules of different configurations were fabricated and tested. Concentration experiments were conducted in a semi-batch mode with a 3-inch spiral-wound module with an effective membrane area of 1.5 m^2. A complete concentration run involved an initial concentration run and two or three successive diafiltration (dilution/reconcentration) runs. Permeation tests were conducted with actual zein extract at various feed pressures to determine the optimum operating pressure at a given feed concentration. Samples of zein concentrate were sent to Illinois Corn Marketing Board (ICMB) and USDA for preparation of spray-dried zein, and analysis and qualitative evaluation of product quality. The spray-dried product was found to have been stripped of most unfavorable odor and taste components.

Membrane fouling remains one of the most challenging obstacles in any membrane technology application. To provide a comprehensive membrane technology process for zein recovery, various cleaning methods and cleaning agents were evaluated to determine the best fit for this application. MTR determined that an enzymatic detergent effectively and safely cleaned fouled modules. Fouled membrane modules cleaned with the detergent recovered their initial fluxes.

Based on the experimental results, several process designs were proposed. The key considerations for the process design were the purity of the zein produced, the final concentration of zein extract, the locations of diafiltration steps, the operating pressure, and the degree of recovery and recycle of the permeated solvent.

SUMMARY OF PLANNED WORK

The project is complete and the final report has been submitted. The next step will be to translate these achievements to a pilot-scale operation to further evaluate the long-term performance and the scalability of the proposed membrane process. Future work should also include the development and evaluation of new membrane materials that have higher sustainable flux and better fouling resistance.

The next step in the process of commercializing the zein extraction/purification technology is to construct the first proof-of-concept demonstration plant. MTR, the University of Illinois, the Illinois Corn Marketing Board (ICMB), and the Washington Group, an engineering and design company that builds plants for the corn milling industry, have formed a consortium with the objective of bringing this technology to the marketplace. Concurrently, the Illinois Corn Marketing Board will sponsor a repeat of the Canadian POS Pilot Systems' bench-scale work with the objective of producing 50 pounds of zein for further evaluation by end-users.

PROJECT ANALYSIS

The project appears to have potential and industry interest. More zein needs to be produced for evaluation in order to generate additional interest. A proof of concept plant would be a great step in proving the technology. Attention appears to be needed in the area of membrane fouling. This project warrants continued attention by DOE.

ACTION REQUIRED BY DOE HEADQUARTERS

No action is required from DOE Headquarters at this time.

STATEMENT OF WORK
DE-FG-36-02GO12057
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Detailed Task Description

Task 1. Prepare Membranes and Modules

Organic-liquid-permeable, stable composite membranes will be prepared. The membrane consists of a finely microporous organic-solvent-resistant polyvinylidine fluoride (PVDF) layer onto which the selective 0.5- to 2.0- μm-thick nonporous polyether/polyamide selective layer is coated. The properties of the membrane will be manipulated by changing the chemistry of the polyether/polyamide layer. Various grades of the polymer are commercially available. For these initial pilot-scale feasibility tests and optimization work, 2.5-inch-diameter membrane modules, containing 1-2 m^2 membrane area will be made.

Task 2. Determine Module Performance with Model Solutions

Modules prepared in Task 1 will first be characterized with simple, easily analyzed one-component model solutions, containing zein, polyethylene glycol (PEG) of various molecular weights, sucrose, raffinose and simple fatty acid esters. The objective of these tests is to show that the membranes and modules can be produced reproducibly and defect-free and to select three or four module types of graded permeability, with properties spanning those required for this process. Tests with mixed zein/raffinose solutions will also be performed to determine the effect of zein on the rejection of lower-molecular-weight compounds such as raffinose.

The selected modules will then be evaluated with zein extracted from corn with 70% ethanol/30% water. Most of the zein extract tests will be performed with zein solutions containing 0.5-1.5 wt% zein. The performance of the membranes in this concentration range is a predicator of membrane performance in the membrane purification part of the zein separation process. However, a few tests with high-concentration zein solutions will be performed to predict membrane performance with 6-12 wt% zein extracts. Based on the test results, the best membranes will be selected for incorporation into the proof-of-concept plant.

Task 3. Prepare Process Design

A preliminary design of the proof-of-concept system to process 2-5 gal/min of zein extract will be prepared. The design will be determined by the relative permeability of the membrane to zein and low-molecular-weight impurities. A simple one-stage process concentrating zein from 0.6 wt% to 6-12 wt% is unlikely to remove enough of the low-molecular-weight impurities, unless the membrane has essentially zero rejection to these components. Some form of diafiltration is likely to be required. In this process, the dilute zein extract is first concentrated 5- to 6-fold, rediluted, and then reconcentrated in a second step. The commercial process simulator ChemCad 5.0 (ChemStations, Inc., Houston, TX) will be used to design the process. MTR's engineering group will prepare the overall process flow diagrams and a P&ID of the proof-of-concept unit.

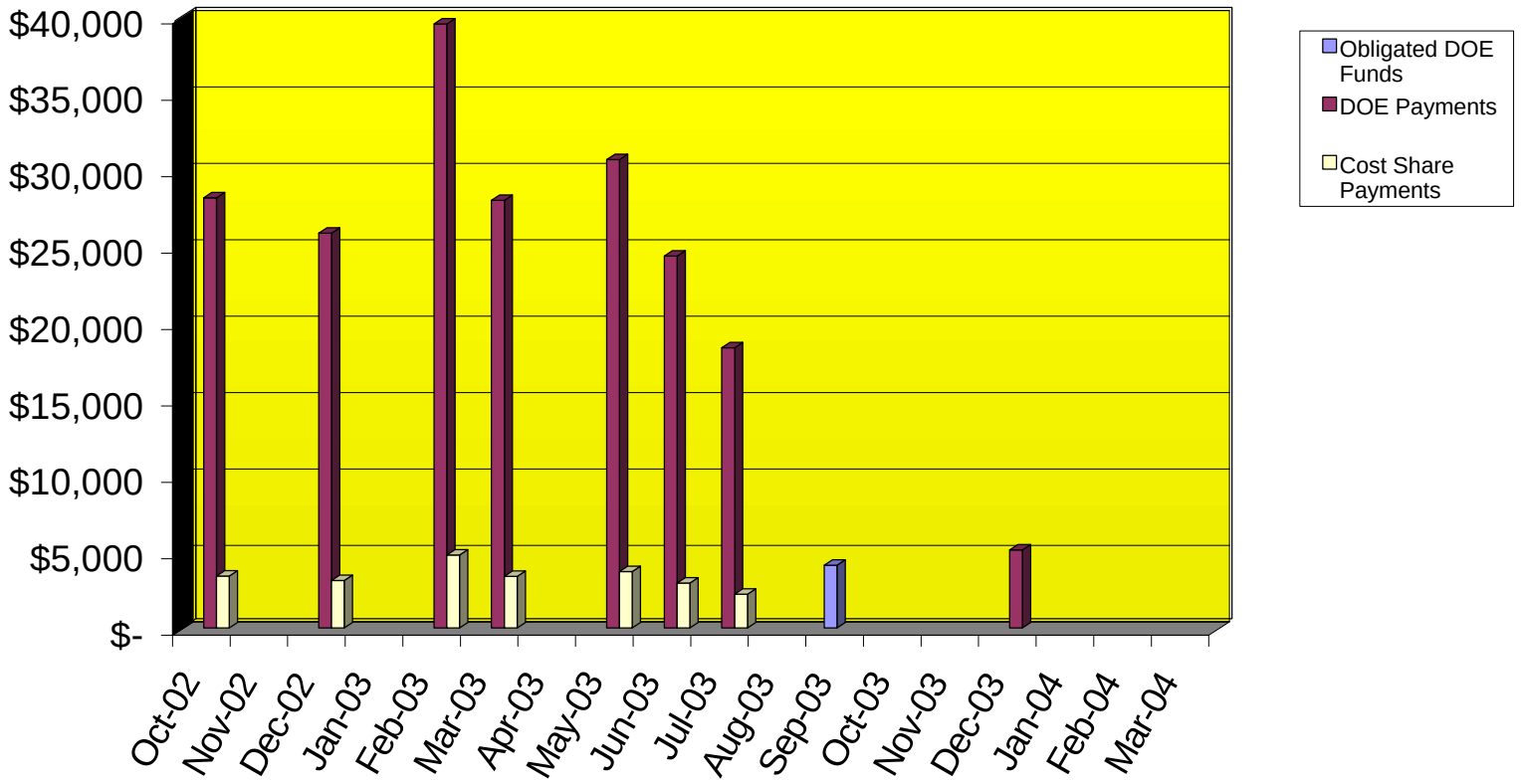
Task 4. Manage Project/File Patents/Prepare Reports

Management of the project will be accomplished by regular weekly meetings attended by Dr. Mairal and the engineers working on the project. Monthly project reports will also be prepared together with a final, detailed project report at the end of the project. MTR has an active program to protect its technology by filling patents as appropriate; the company's in-house patent agent will make this determination and the cost will be borne by MTR.

Project Cost Performance in DOE Dollars for Fiscal Year 2003

DE-FG36-02GO12057 Membrane Technology & Research, Inc.

Separation of Zein from Bioethanol Process Streams



	Oct-02	Nov-02	Dec-02	Jan-03	Feb-03	Mar-03	Apr-03	May-03	Jun-03	Jul-03	Aug-03	Sep-03
Obligated DOE Funds	$0	$0	$0	$0	$0	$0	$0	$0	$0	$0	$0	$4,117
DOE Payment	$28,154	$0	$25,845	$0	$39,543	$27,999	$0	$30,667	$24,349	$18,341	$0	$0
Cost Share Payment	$3,400	$0	$3,121	$0	$4,775	$3,381	$0	$3,703	$2,940	$2,215	$0	$0

	Oct-03	Nov-03	Dec-03	Jan-04	Feb-04	Mar-04	PFY*	Cumulative
Obligated DOE Funds	$0	$0	$0	$0	$0	$0	$195,883	$200,000
DOE Payment	$0	$0	$5,104	$0	$0	$0	$0	$200,000
Cost Share Payment	$0	$0	$0	$0	$0	$0	$0	$23,537

Approved DOE Budget:	$200,000
Approved Cost Share Budget:	$24,153
Total Project Budget:	$224,153

* Prior Fiscal Years

Membrane Technology & Research - 02GO12057

ID	Task Name	Start	Finish
1	Prepare Membranes and Modules	Fri 8/9/02	Fri 11/8/02
2	Module Performance with Model Solutions	Mon 11/11/02	Tue 3/11/03
3	Prepare Process Design	Mon 10/7/02	Fri 6/6/03
4	Manage Project/File Patents/Prepare Reports	Fri 8/9/02	Fri 8/29/03